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Q 79161



INVESTOR IN PEOPLE

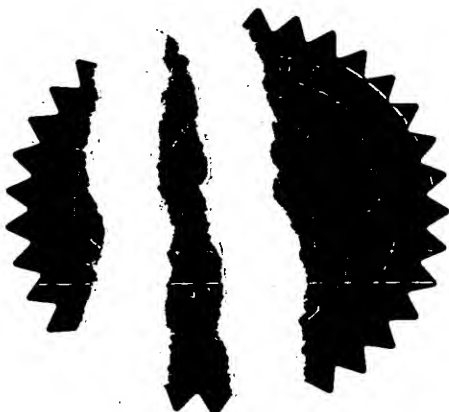
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In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

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Signed

*He Behen*

Dated 25 November 2003

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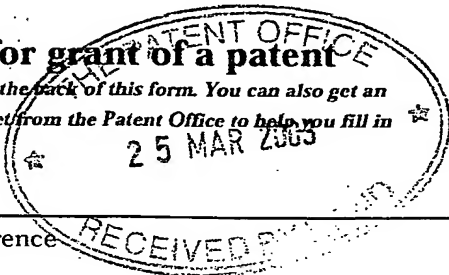


The  
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1/77  
26 MAR 03 E795031-5 002890  
P01/7700 0.00-0306829.3

# Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)



The Patent Office

Cardiff Road  
Newport  
South Wales  
NP9 1RH

1. Your reference PJF01560GB

2. Patent application number 0306829.3  
(The Patent Office will fill in this part) 25 MAR 2003

3. Full name, address and postcode of the or of each applicant (underline all surnames)

Oxford Diffraction Ltd.  
20 Nuffield Way  
Abingdon  
Oxfordshire, OX14 1RL  
UK

Patents ADP number (if you know it)

8596876001

If the applicant is a corporate body, give the country/state of its incorporation UK

4. Title of the invention HIGH FLUX X-RAY SOURCE

5. Name of your agent (if you have one) Gill Jennings & Every

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

Broadgate House  
7 Eldon Street  
London  
EC2M 7LH

Patents ADP number (if you know it) 745002 ✓

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number	Country	Priority application number (if you know it)	Date of filing (day / month / year)

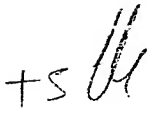
7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application	Number of earlier application	Date of filing (day / month / year)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

- a) any applicant named in part 3 is not an inventor, or
  - b) there is an inventor who is not named as an applicant, or
  - c) any named applicant is a corporate body.
- See note (d))

# Patents Form 1/77

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Claim(s)	2
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10. If you are also filing any of the following, state how many against each item.

Priority documents	-
Translations of priority documents	-
Statement of inventorship and right to grant of a patent (Patents Form 7/77)	0
Request for preliminary examination and search (Patents Form 9/77)	1
Request for substantive examination (Patents Form 10/77)	0
Any other documents (please specify)	NO

11. For the applicant  
Gill Jennings & Every

I/We request the grant of a patent on the basis of this application.

Signature

Date

25 March 2003

12. Name and daytime telephone number of person to contact in the United Kingdom

FINNIE, Peter John

020 7377 1377

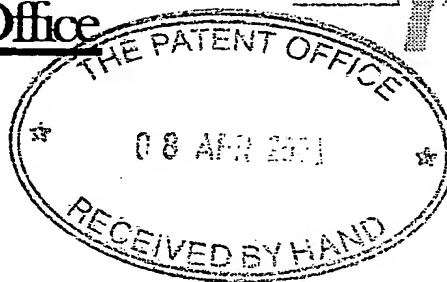
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## Notes

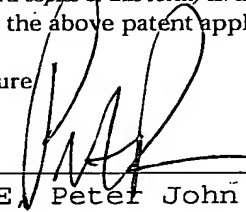
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# Statement of inventorship and of right to grant of a patent



The Patent Office

Cardiff Road  
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NP9 1RH

1. Your reference	PJF01560GB
2. Patent application number (if you know it)	0306829.3
3. Full name of the or of each applicant	Oxford Diffraction Ltd.
4. Title of the invention	HIGH FLUX X-RAY SOURCE
5. State how the applicant(s) derived the right from the inventor(s) to be granted a patent	By employment.
6. How many, if any, additional Patents Forms 7/77 are attached to this form? (see note (c))	0
7. For the applicant Gill Jennings & Every	<p>I/We believe that the person(s) named over the page (and on any extra copies of this form) is/are the inventor(s) of the invention which the above patent application relates to.</p> <p>Signature  Date 8 April 2003</p>
8. Name and daytime telephone number of person to contact in the United Kingdom	<p>FINNIE, Peter John</p> <p>020 7377 1377</p>

## Notes

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Enter the full names, addresses and postcodes of the inventors in the boxes and underline the surnames

KUCHARCZYK, Damian  
Stawowa 3/2  
PL-50-018  
Wroclaw  
Poland

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05607343001

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## HIGH FLUX X-RAY SOURCE

### Field of the Invention

X-ray diffraction is an analytical technique for obtaining information on the structure of atoms or molecules in a crystal from the diffraction pattern that is produced by diffraction of rays by the crystal's atomic planes. The present invention generally relates to a high flux X-ray source for use in X-ray diffraction techniques for analysing specimens by irradiating with X-rays and, more particularly, to a high flux X-ray source of a type utilising an artificial multi-layered optic with a sealed X-ray tube.

### Background to the Invention

X-ray sources currently utilised in the field of single crystal X-ray diffraction comprise two main types. A first type, the sealed X-ray tube, is traditionally utilised where a metal target (anode) typically of a metal such as molybdenum, copper or silver is bombarded with electrons from a tungsten filament and mounted within a vacuum. A sealed ray tube is commonly used in combination with a graphite monochromator and a collimator and is typically run up to a maximum power of 2-3kW as the anode will not support a much greater heat load without being destroyed. This heat is dissipated through water cooling of the anode. In order to realise a greater intensity X-ray source, a greater power density of electrons needs to be placed on the anode. To achieve this, a second type of source, the rotating anode, was developed. In a rotating anode source, the anode consists of a rotating water cooled drum, the external surface of which has been deposited with the metal target. The drum sits in a high vacuum enclosure and the rotating anode is run at powers of up to 18 kW, although a more typical power is 5 kW.

The sealed X-ray tube has the advantage that the apparatus for its use is compact, reliable, and easy to use and maintain. The extent of maintenance is generally limited to the change over of a burnt out X-ray tube, which typically last 10,000 hours. Change over and alignment of the replacement X-ray tube in a traditional sealed tube set-up can take in the region of 1-2 hrs. Recent improvements to the traditional set-up, which utilise specific geometries have greatly reduced this change over and alignment time to approximately 10-30 mins. By comparison the rotating anode which can be up to 10 times larger, requires much greater maintenance and attention. In particular, the electron producing filament in the rotating anode

requires change over on burn out typically ever 6 weeks, and the rotating anode drum requires replacement at least every 6 months. In each, case the changeover and required re-alignment can take a total of 3-8 hrs.

5 A comparison of the x-ray intensity out put can be obtained from a simple appreciation of the classical formula describing the relationship between the intensity  $I_K$  of  $K_\alpha$  X-rays produced by an X-ray source and the operating voltage  $V$  (in kV) and current  $i$  (in mA)

$$I_K = iB(V - V_K)^{1.5}$$

10

where  $V_K$  is the excitation voltage for K series radiation in kV and  $B$  is a constant of proportionality. By way of example, a rotating anode running at 5kW (typically 50kV and 100 mA) only generates in the region of 2.5 times the X-ray intensity generated by a sealed tube running at 2kW power (typically 50 kV and 40mA), since the term  $B(V - V_K)^{1.5}$  is identical in each case.

15

The rotating anode source is often used in combination with optics, including mirrors. Reports in both the literature and patent applications concerning such systems are numerous, and detail the construction of the optics in terms of both the deposition of layers of specific chemical materials and the optimisation of specific shapes which serve to focus X-rays, as well as the specific distances and angles required for optimal performance of the optics. The most successful of the optics produced to date are the multi-layer optics, which have enabled a great increase in the usable X-ray intensity through the focussing of a large amount of the X-rays emitted from the source.

20

25 Compact X-ray sources which use a sealed X-ray tube could also benefit from the use of appropriate optics. The efficiency of conversion of usable X-rays from a sealed X-ray tube is typical 1%. But by using suitable optics, including mirrors, it is theoretically possible to harvest a greater proportion of these X-rays and to focus them tightly to provide an increased level of usable X-ray intensity. Furthermore it has been indicated that the use of multi-layer optics has the ability to increase the flux from a fine focus sealed X-ray tube by more than an order of magnitude over that obtained with total reflection mirrors. However, depending on design and configuration, such optics may increase complexity, reducing the ease of maintenance of the source and reducing the benefits of employing a sealed tube.

30



There are currently no X-ray sources that combine higher performance optics with a sealed X-ray tube to achieve X-ray flux levels approaching those obtainable from the much larger rotating anode based systems. There is therefore a need for a compact X-ray source which can deliver the flux levels required for numerous applications, including the irradiation of samples in life science investigations, but which has the appropriate ease of maintenance and enables simple re-alignment when replacement of the sealed X-ray tube is required.

### Summary of the Invention

According to one aspect of the present invention, a high flux X-ray source comprises:

- a sealed X-ray tube;

- an optic housing containing a multi-layer optic for collecting and focussing X-rays generated in the sealed X-ray tube, wherein the multi-layer optic is located at a predetermined distance from the sealed X-ray tube and the optic housing is adjustable relative to the sealed X-ray tube; and,

- an X-ray beam conditioner, the beam conditioner being adjustable relative to the optic housing.

The use of a multi-layer optic provides for the efficient collection and focussing of X-rays generated in a compact sealed tube. The wavelength dependence of a multi-layer optic also enables it to act as a monochromator, providing a beam of X-rays with a predetermined range of photon energy.

Preferably, a multi-layer optic comprises a confocal mirror. Alternatively, the multi-layer optic may comprise a sagittal mirror. The multi-layer optic may be glued into position within the optic housing.

Preferably, the optic housing is adjustable relative to the sealed X-ray tube by independent rotation in each of a first pair of substantially orthogonal directions.

Preferably, the optic housing is also adjustable relative to the sealed X-ray tube by independent translation in each of a second pair of substantially orthogonal directions.

The first pair and second pair of orthogonal directions may be coincident. Alternatively, they may be orientated at an angle to one another, including 45°.

The use of orthogonal axes of adjustment affords quick and easy alignment of the optic relative to the direction of the X-rays emerging from the sealed X-ray tube

and may also offer control over the size and intensity of the X-ray focal spot.

The presence of an X-ray beam conditioner provides for additional collection and beam shaping of the X-rays together with further directional control of the X-ray beam onto a particular location or target sample. The beam conditioner may take a number of forms, including that of an X-ray collimator, according to the desired final spatial form of the X-ray beam.

Preferably, the X-ray beam conditioner is detachable.

Preferably, the X-ray beam conditioner is adjustable relative to the optic housing by independent tilt controls in each of a pair of substantially orthogonal directions.

Preferably, the X-ray source further comprises a shutter housing located between the sealed X-ray tube and the optic housing.

Preferably, the shutter housing is adapted to receive a portion of the optic housing.

This arrangement allows simple and consistent alignment of the optic housing relative to the sealed X-ray tube during both first assembly of the source and reassembly of the source during replacement of the sealed X-ray tube, for example.

Preferably, the optic housing is flooded with an inert gas to reduce damage to the multi-layer optic and to reduce scattering of the X-rays during propagation through the optic housing. Preferably, the inert gas is helium.

Preferably, the X-ray source further comprises a moveable X-ray beamstop. Preferably, the beamstop is located beyond the X-ray beam focus.

A moveable X-ray beamstop allows for the safe termination of the X-ray beam during operation whilst also providing the facility to displace the beamstop for easy placement of a target sample prior to operation.

According to another aspect of the present invention, X-ray irradiation apparatus comprises an X-ray source according to the one aspect of the present invention and means for holding a sample in the path of the focussing X-rays.

Thus the present invention addresses the need for a compact high flux X-ray source by combining sealed X-ray tube technology with high performance multi-layer optics contained within a housing that is easily located relative to the X-ray tube and can be finely adjusted for optimum collection and focussing of the X-rays. The multi-layer optics may also serve as a beam monochromator. A compact adjustable beam conditioner, which may be a collimator, ensures that the X-ray beam has the desired

final spatial form for a given application.

### **Brief Description of the Drawings**

5 An example of the present invention will now be described in detail with reference to the accompanying drawings, in which:

Figure 1 shows a perspective view of the high flux X-ray source with detached conditioner and the X-ray beamstop rotated by 90°.

Figure 2 shows a perspective view of the high flux X-ray source and the adjustment screws for alignment of the multi-layer optics and beam conditioner;

10 Figure 3 shows a side view and a plan view of the high flux X-ray source;

Figure 4 shows a side view of the high flux X-ray source and cross-sections B-B and C-C through the X-ray source; and,

Figure 5 shows a perspective view of the high flux X-ray source mounted on a stand with screws for final directional adjustment.

15

### **Detailed Description**

Figure 1 shows an embodiment of the present invention, which applies a novel optical arrangement of a sealed X-ray tube with a multi-layer mirror optic to the construction of a high flux X-ray source 1 for increased efficiency of collection and focussing of X-ray photons generated in the sealed X-ray tube. The construction of the high flux X-ray source comprises an X-ray tube shield 2 containing the sealed X-ray tube, a fast X-ray shutter located in a shutter housing 3, a multi-layer optic housing 4 containing a multi-layer optic, an X-ray beam conditioner or collimator 5, X-ray beam stop 6 and a counter weight 7 for counterbalancing the weight of the beam directing portion of the source when mounted on a stand. The nozzle region 8 may further contain a  $K_{\beta}$  filter for filtering out residual unwanted  $K_{\beta}$  radiation. As indicated, the X-ray beam conditioner or collimator 5 section is detachable from the rest of the source.

25 The X-ray source has been designed to be of a compact construction, occupying a footprint in the region of 0.15m<sup>2</sup> or less. Therefore, an example of the approximate overall dimensions might be 0.32×0.46×0.08m. Furthermore, the source has been constructed with a minimum number of adjustments to enable ease of use and maintenance, with a minimum of downtime. The source has been designed with use in small molecule crystallography and life science applications particularly in mind, where the irradiation of samples would otherwise require the X-ray intensity provided

30

by a rotating anode or synchrotron X-ray source.

The X-rays are generated in the copper anode sealed tube and emitted through the tubes' beryllium window before passing through a hole in the X-ray tube shield 2. This hole is closed by a moveable shutter located in the shutter housing 3.

5 When the shutter is open the X-rays pass through a helium flooded path and at a critical distance the X-rays are collected and focused by a multi-layer optic within the multi-layer optic housing 4, the position of which can be fine tuned to affect the size and intensity of the X-ray focal spot. The focused X-ray beam then passes along the collimator or beam conditioner 5 and may pass through a Ni foil  $K_{\beta}$  filter, and/or a kapton window. At this point the X-ray beam passes into air and is focussed at a point where a sample would be placed for irradiation. A proportion of the X-ray beam continues straight through the sample and falls on the moveable lead packed beamstop 6. The X-ray beamstop 6 has been designed to permit rotation through 90 degrees to provide greater accessibility for mounting of the sample for irradiation. 10 Once the optics are aligned then a maximal X-ray intensity should have been achieved. These X-rays then need to be directed in the correct direction, which is onto the sample position. This is achieved by five adjustment screws located on the base of the assembly.

We now consider the alignment and construction of the source in more detail with reference to Figures 2 to 4. The X-ray tube typically utilised is a ceramic long fine focus 2kW sealed tube of copper anode and is inserted into the brass tube shield from the front of the X-ray source. The X-ray tube shield, has a circular hole critically placed to allow the X-rays generated from the X-ray tube and emitted through the tube's beryllium window to be collected and passed to the multi-layer optic. Directly 25 attached to the right hand-side of the tube shield is the X-ray fast shutter housing located in a 1mm recess in the external surface of the X-ray tube shield.

The X-ray shutter consists of a piece of lead attached to one end of a moveable arm the other end of which is attached to a high precision stepper motor of high speed response time, typically 3 milliseconds, the shutter can thus be moved 30 over a range of typically 6 degrees. The shutter is electronically controlled and its status can be constantly monitored. The shutter can be easily interchanged and is secured in place by the screw 14. Releasing this screw the shutter assembly can be withdrawn from the rear of the X-ray shutter housing. On the side parallel to the aperture in the X-ray housing is an aperture for location of the optic housing unit. The

optic housing is inserted into this aperture and extends inside the X-ray shutter housing. An X-ray leak proof coupling is afforded by a flexible washer assembly.

Extending through the X-ray shutter housing are the two adjustment screws 5(1) and 5(2). These serve to apply pressure to the optic housing where it extends  
5 inside the X-ray shutter housing. By adjustment of these screws pressure can be applied to the optic housing in the x and y directions and the optic housing thus caused to pivot on the vertical and horizontal pivot screws 3 and 4. Release of the screws 5(1) and 5(2) causes the X-ray optic housing to return to its previous position due to two compression springs which are mounted in line with the screws 5(1) and  
10 5(2). The screw 4 serves as both a pivot point for the vertical direction and an adjustment screw in the horizontal (x) plane. Tightening of screw 4 applies pressure to the X-ray optic housing and affords positional movement of the X-ray optic housing in the horizontal (x) plane, which is caused to pivot on the screw 3. Release of screw 4 affords positional movement of the X-ray optic housing in the horizontal (x) plane  
15 and in the direction of the screw due to a tension spring.

Screw 2 is one of three grub screws which serve to hold in place the detachable collimator / beam conditioner assembly. Internally this is sealed by an O-ring to prevent leakage of helium gas at this point. The aperture of the beam conditioner is adjustable in x and y directions through the use of screws 7 and 8. On  
20 screwing in screw 7 or 8, pressure is applied to the beam conditioner causing it to tilt and thus reposition the final aperture. On releasing the screws, the movement is reversed due to compression springs mounted in line with the screws. The final aperture is contained in a removable end cap 6, which allows for this to be exchanged for various sizes. The end of the beam conditioner in the X-ray beam path is sealed  
25 with a kapton window and the helium gas exits by another path close to the final aperture.

The internal construction of the present invention is such that the X-ray optic housing extends to a position close to the X-ray aperture in the X-ray tube shield. In the intervening space is mounted the X-ray shutter lead block. The end of the X-ray  
30 optic housing nearest the X-ray tube is closed with a kapton window, with the other end open. Within the X-ray optic housing are mounted the multi-layer optics, which are in a fixed position. The optics may be affixed to the wall of the housing by glue. Either a single multi-layer optic or a combination of multi-layer optics may be used. Multi-layer optics are usually constructed from several layers of lighter and heavier

materials giving rise to a distributed reflecting structure. The structure may even be graded. Examples of suitable optics include the confocal mirror and the sagittal mirror. The confocal mirror typically comprises two extended multi-layer reflectors at right angles, and so the X-rays collected undergo a double bounce as they propagate through the optic housing. Due to the wavelength Bragg selectivity of the reflector, the double reflection can act as an efficient monochromator, providing a substantial filtering of unwanted X-ray wavelengths and reducing the need for a separate filter.

The X-ray beam path through the present invention is maintained under a constant flow of helium gas which is supplied via the helium entry port 13 and fed internally along a narrow diameter tube which opens into to the X-ray optic housing at a point close to the kapton window at the X-ray tube end. The presence of helium serves to reduce possible damage to the sensitive multi-layer optics which are subjected to X-rays and also to provide the maximum X-ray intensity to be placed on the sample. X-ray intensity is reduced with distance from the source as a result of scatter from the air. This reduction of intensity is minimised by replacing air with helium.

The counterweight consists of a lead filled block which is secured on the left side of the X-ray tube shield and serves to counterweight the combined weight of the X-ray shutter, X-ray shutter housing, X-ray optic housing, collimator / beam conditioner and beamstop assembly which are mounted on the right of the X-ray tube shield. A screw 1 extends through the counterweight to allow a front face panel to be mounted to cover the adjustment screws 5(1) and 5(2) and the base of the X-ray tube.

The X-ray beamstop mount incorporates an X-ray warning lamp which is connected via an external cable to the electronics of the fast X-ray shutter. When the shutter is open and X-rays are exposed the warning lamp is illuminated. The beamstop needs to be aligned so that the lead packed tip is aligned in the X-ray beam. Alignment of the beamstop is achieved in the horizontal (x) direction using the screw 11 and in the vertical (y) direction using screw 10. The beamstop can be rotated through 90 degrees by pressure applied in the region of the screw 10, via a thumb hold located on the front of the device. A reproducible position of the beamstop is achieved as the beamstop incorporates a spring loaded ball bearing which locates in sockets at the 'up' and 'down' position of the beamstop, these positions being at 90 degrees to each other. On screwing in screw 10 or 11 the movement causes the ball bearing spring to be indirectly compressed. The 'down'

socket is designed with a gradient on one side, which causes the spring compression to increase as screw 11 is screwed in. On releasing screw 10 or 11 the spring force acting on the ball bearing resting on the gradient causes the beam stop to move in the reverse direction. The user may rotate the beam stop to the 'up' position to facilitate sample handling and after returning to the 'down' position the previously aligned position is automatically re-established. The entire beam stop may be detached by removing screw 12. The distance of the beam stop from the location of the sample is adjustable on a slider, which is locked with screw 9.

Figure 5 illustrates the X-ray source mounted on a stand, with the counterweight serving to counterbalance the weight of the beam directing portion of the source. The complete source assembly can be pointed in the correct horizontal and vertical position using five screws located on the base. Three of the screws, one at the front 1 and two at the back 3(1) and 3(2), adjust the vertical position by applying pressure to the underside of the X-ray tube shield. The remaining two screws 2(2) and 2(1) can be adjusted against each other so to apply pressure to the back left and right hand sides of the X-ray tube shield, respectively. These two screws afford movement of the complete assembly in the horizontal plane. Thus, once the source has been optimised for maximum X-ray flux concentration by adjustment of the multi-optic housing and beam conditioner/collimator, the beam can be accurately targeted on a sample by adjustment of the base screws.

## CLAIMS

1. A high flux X-ray source comprises:  
a sealed X-ray tube;  
5 an optic housing containing a multi-layer optic for collecting and focussing X-rays generated in the sealed X-ray tube, wherein the multi-layer optic is located at a predetermined distance from the sealed X-ray tube and the optic housing is adjustable relative to the sealed X-ray tube; and,  
an X-ray beam conditioner, the beam conditioner being adjustable relative to  
10 the optic housing.
2. An X-ray source according to claim 1, wherein the multi-layer optic is a confocal mirror.
- 15 3. An X-ray source according to claim 1, wherein the multi-layer optic is a sagittal mirror.
4. An X-ray source according to any of claims 1 to 3, wherein the optic housing is adjustable relative to the sealed X-ray tube by independent rotation in each of a first  
20 pair of substantially orthogonal directions.
5. An X-ray source according to claims 1 to 4, wherein the optic housing is adjustable relative to the sealed X-ray tube by independent translation in each of a  
25 second pair of substantially orthogonal directions.
6. An X-ray source according to any preceding claim, further comprising a shutter housing adapted to receive a portion of the optic housing.
7. An X-ray source according to any preceding claim, wherein the optic housing  
30 is filled with an inert gas.
8. An X-ray source according to claim 7, wherein the inert gas is helium.



9. An X-ray source according to any preceding claim, further comprising a moveable X-ray beam stop.

5 10. X-ray irradiation apparatus comprising an X-ray source according to any preceding claim, and means for holding a sample in the path of the focussing X-rays.

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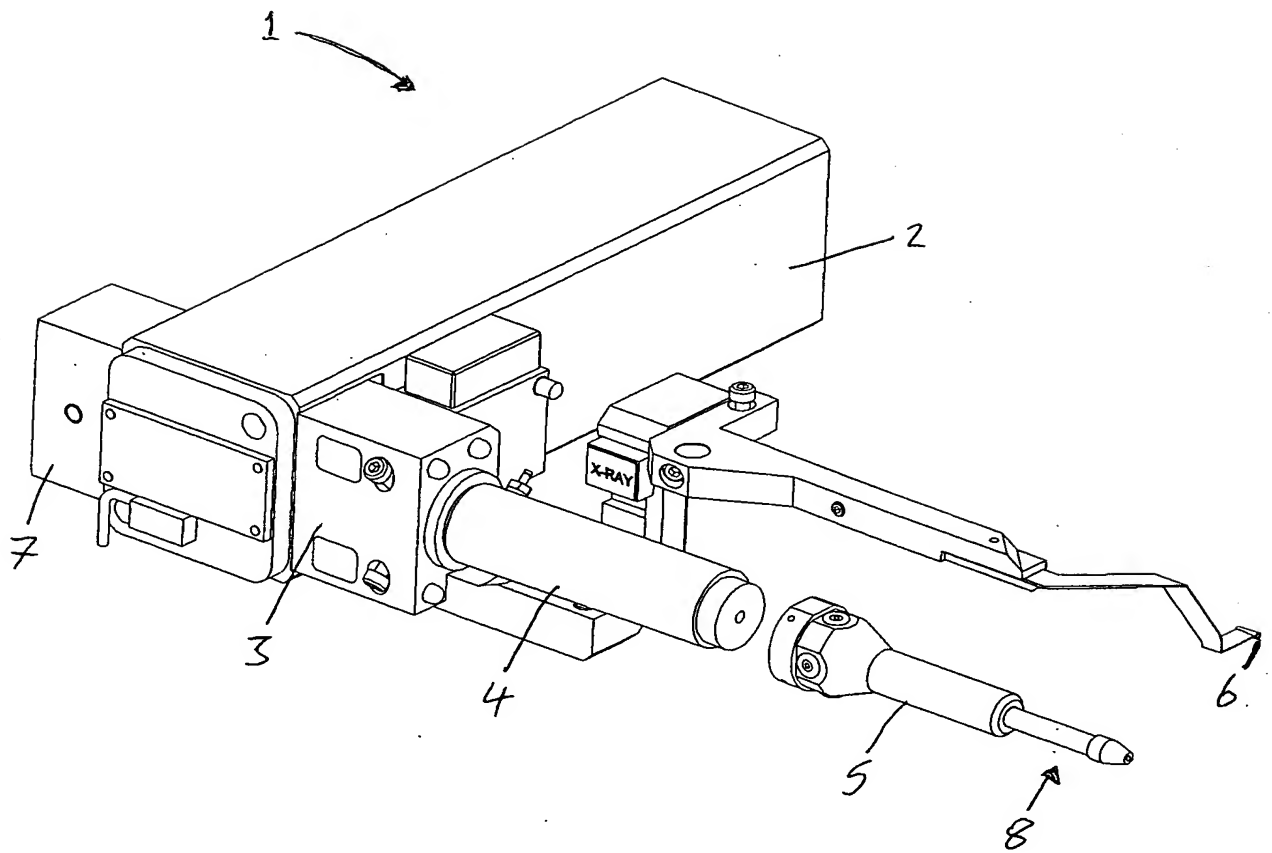


Figure 1

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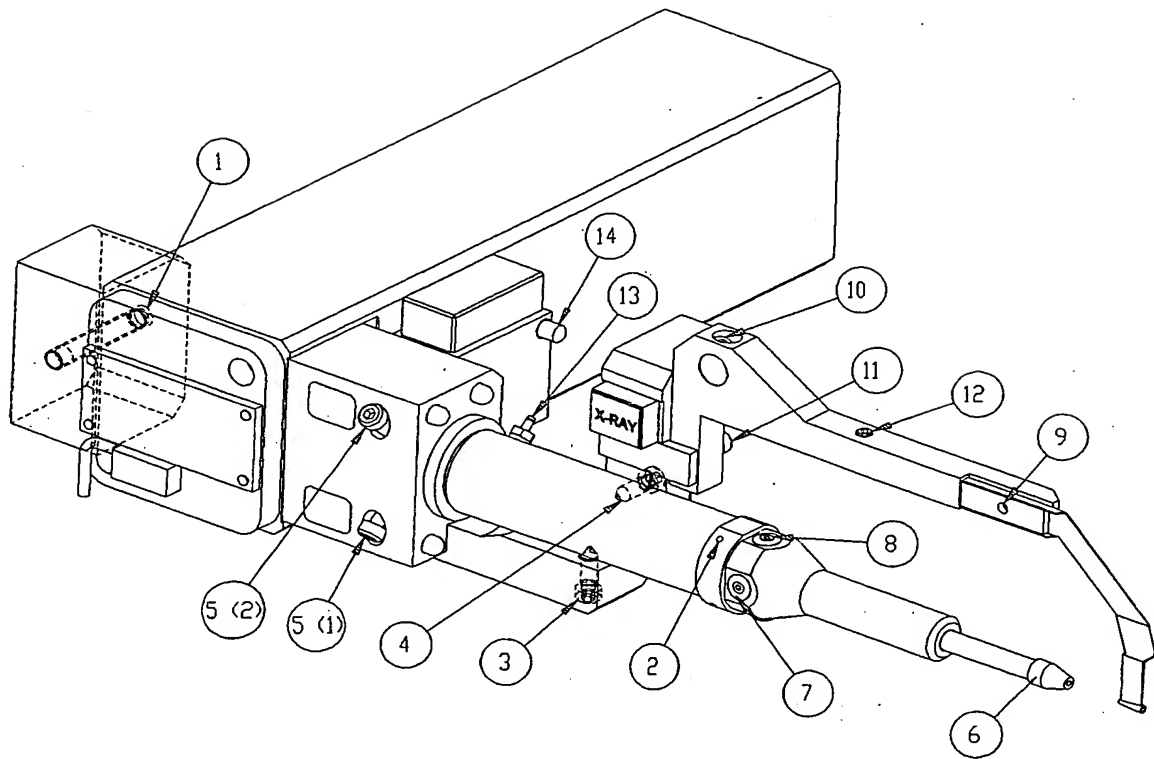


Figure 2

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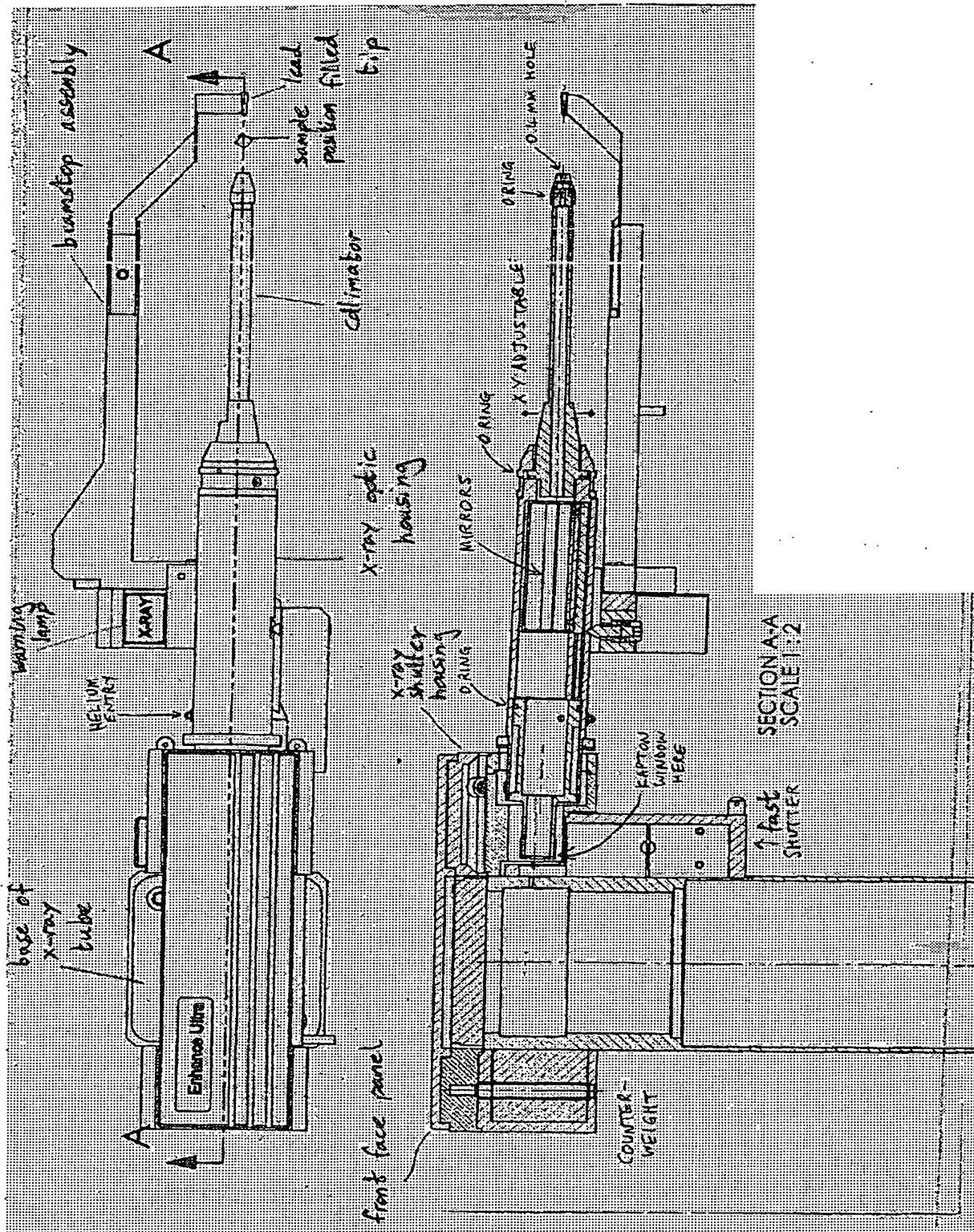


Figure 3

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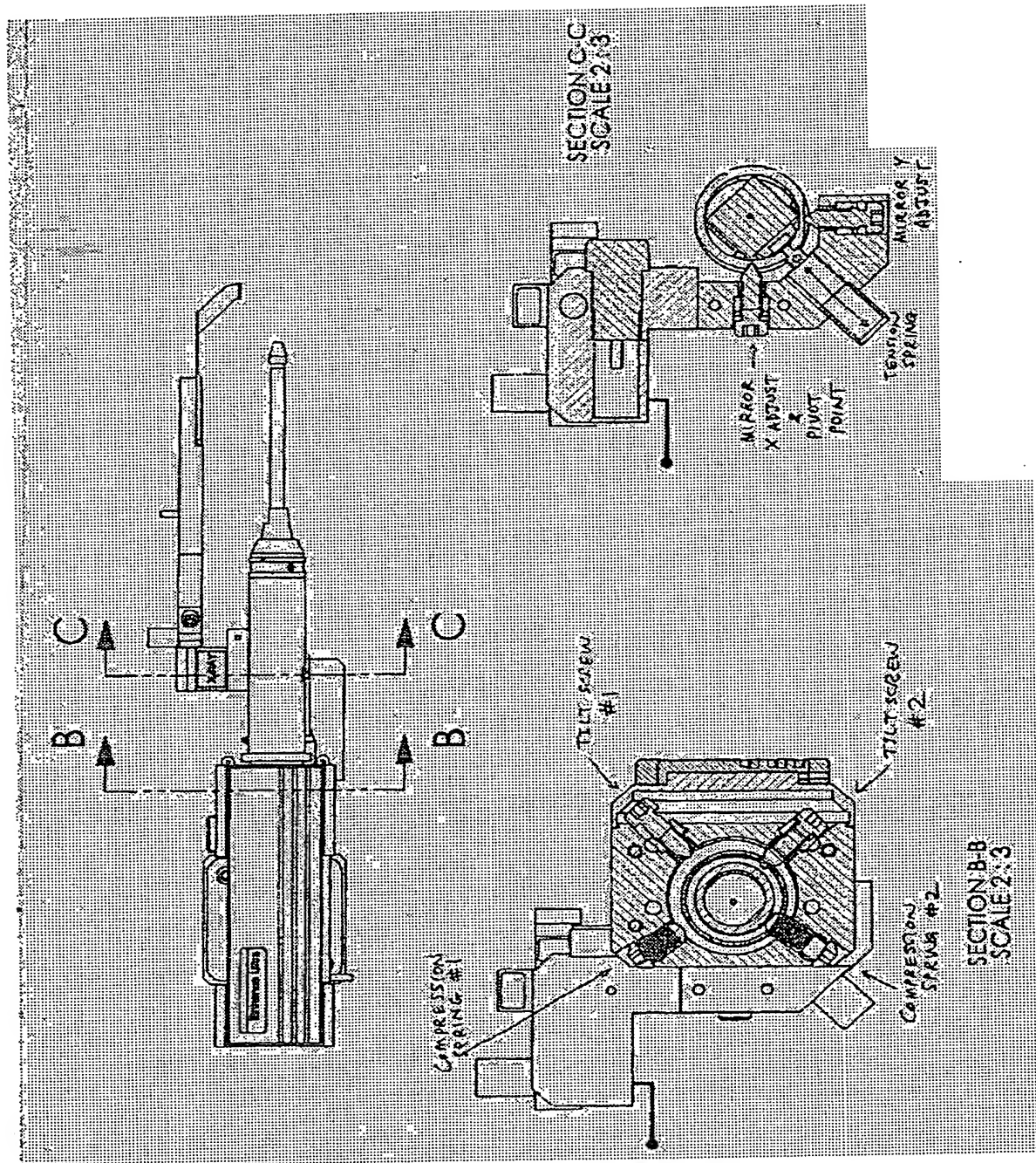


Figure 4

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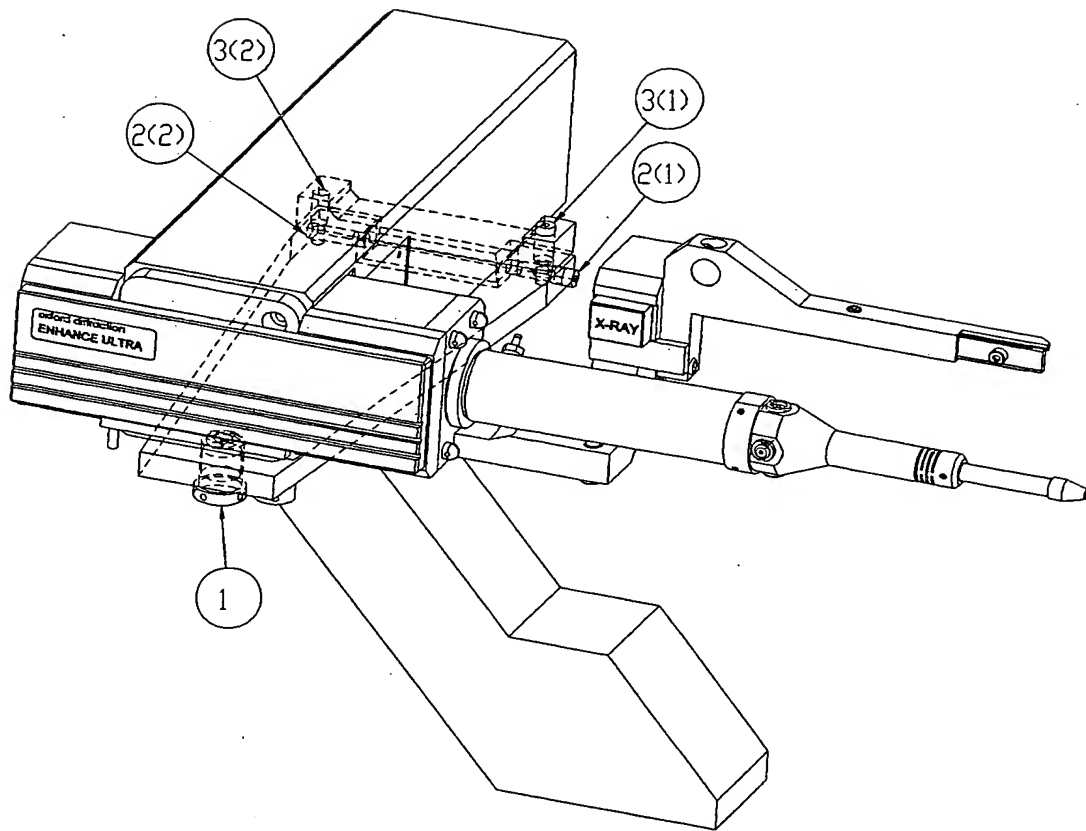


Figure 5

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